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EVALUATION OF BIOLOGICAL TREATMENT

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Cover photo: Compost piles on the compositing area of Metsä-Tuomela waste treatment station
Photo: Jukka Puustinen

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Jukka Puustinen, Kirsten S. Jørgensen, Tapio Strandberg and Anna-Mari Suortti

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Abstract

Biological treatment of contaminated soil has received much attention during the last decade. Microbes are known to be able to degrade many oil hydrocarbons. However, research is needed to ensure that new technologies are implemented in a safe and reliable way under Finnish climatic conditions. The main points of interest are the rate of the degradation as well as the survival and efficiency of microbial inoculants possibly introduced during the treatment.

During 1993 the biotreatability of oil-contaminated soil from service stations was investigated in cooperation with the Finnish Petroleum Federation. The goal of this field-scale study was to test how fast lubrication oil can be composted during one Finnish summer season and to find out whether microbial inoculants would enhance the degradation rate.

The soil was excavated from three different service stations in the Helsinki metropolitan area and was transported to a controlled composting area. The soil was sieved and compost piles, also called biopiles, were constructed on the site. Bark chips were used as the bulking agent and nutrients and lime were added to enhance the biological activity. Two different commercial bacterial inoculants were added to two of the piles. The piles were turned by a tractor-drawn screw-type mixer at two to four weeks interval. Between the mixings, the piles were covered with tarpaulins to prevent evaporation and potential excessive wetting.

Several microbiological parameters were determined during the test period as well as the temperature and mineral oil content. The mineral oil degradation rate was most rapid during the first month, which correlated well with the total microbial activity measured as respiration. The highest temperature measured during the treatment was 44°C.

During 5 months of composting the mineral oil concentration decreased in all the piles from approximately 2400 mg to 700 mg of mineral oil per kg d.wt. of soil. There was no major effect of the added inoculants. In one pile, which did not receive any nutrients, the degradation was delayed about one month but the pile reached the same level of mineral oil at the end of the test as the other piles.

The expences for this experimental composting, without the soil excavation work and transportation, were on average 100 FIM/m^3 contaminated soil. According to the information from contractors this also represents the real market price of the work.

Composting can thus be regarded as an effective and fairly moderate price method for remediating oil contaminated soil.

Keywords
Bioremediation, service station, contaminated soil

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Tiivistelmä
Biologisten keinojen käyttö maan puhdistuksessa on lisääntynyt viimeisten vuosien aikana. Mikrobien tiedetään pystyvän hajottamaan useita haitallisia yhdisteitä maassa. Tarvitaan kuitenkin tutkimusta varmistamaan, että käytetyt biotekniikat ovat järkeviä ja luotettavia Suomen olosuhteissa. Tärkeimpiä selvitettäviä asioita ovat hajotusnopeus, mahdollisten haitallisten hajoamistuotteiden synty, lisättyjen mikrobien säilyminen ja tehokkuus käsittelyn aikana ja sen jälkeen.

Vuonna 1993 tutkittiin huoltoaseman likaantuneen maan biopuhdistusta yhteistyössä Öljyalan keskusliiton kanssa. Kenttäkokeiden tarkoituksena oli selvittää, kuinka nopeasti jäteöljyllä likaantunutta maata voidaan puhdistaa kompostoimalla yhden kesäkauden aikana ja onko lisätyistä mikrobeista hyötyä hajotustoiminnalle.

Likaantunut maa kaivettiin kolmelta Helsinki-seudun huoltoasemalta ja kuljetettiin tarkoitukseen varatulle kompostointikentälle. Maa seulottiin ja siitä rakennettiin aumat kuorikkeen kanssa. Aumoihin lisättiin ravinteita ja niiden pH säädettiin kalkilla. Kahdessa aumassa kokeiltiin kaupallisia mikrobivalmisteita. Aumat pidettiin peitettyinä ja ne käännettiin ruuvityyppisellä sekoittimella kahden ja neljän viikon välein.

Kokeen aikana määritettiin aumamassojen mineraaliöljyjen pitoisuus, lämpötila ja seurattiin useita mikrobiologisia tekijöitä. Mineraaliöljyjen hajoaminen oli nopeinta ensimmäisen kuukauden aikana. Tämä näkyi hyvin myös alkujakson korkeana mikrobiaktiivisuutena. Korkein mitattu lämpötila aumoissa oli 44 °C.

Viiden kuukauden aikana aumojen mineraaliöljypitoisuudet alenivat 2400 mg:sta 700 mg:an kilossa kuivaa maata. Lisätyillä mikrobeilla ei ollut merkittävää vaikutusta hajotukseen. Yhdessä aumassa, johon ei lisätty ravinteita, hajotus oli alussa hitaampaa, mutta mineraaliöljyjen pitoisuudet alenivat lopussa muiden tasolle.

Tutkimuksen pohjalta arvioidut kustannukset kompostoinnille, ilman maansiirtotöitä, ovat keskimäärin noin 100 mk/m^3 likaista maata.

Asiasanat (avainsanat)
Biologinen kunnostus, huoltoasemat, maaperä

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Referat
Under de senaste åren har utnyttjandet av biologiska metoder i marksanering ökat. Det är känt att mikrober förmår nedbryta ett flertal skadliga föreningar i marken. Det behövs dock forskningsinsatser för att säkra att den tillämpade biotekniken är rimlig och tillförlitlig i finländska förhållanden. De viktigaste faktorerna som kräver utredning är nedbrytningshastighet, uppkomst av möjliga skadliga nedbrytningsprodukter, beständighet av tillsatta mikrober samt effektiviteten under och efter behandlingen.

Biorening av förorenad mark vid servicestationer undersöktes år 1993 i samarbete med Oljebranschens Centralförbund. Avsikten med fältförsöken var att påvisa hur snabbt det går att rena mark som förorenats med spillolja genom att kompostera marken under en sommar, samt huruvida tillsatta mikrober främjar nedbrytningen.

Den förorenade marken togs från tre servicestationer inom huvudstadsregionen och transporterades till ett komposteringsfält reserverat för ändamålet. De silade jordmassorna blandades med barkflis och formades till dösar. Näringsämnen tillsattes dösarna, och deras pH reglerades med kalk. I två dösar prövades kommersiella mikrobpreparat. Dösarna hölls täckta och de vändes med en omblandare av skruvtyp med två och fyra veckors mellanrum.

Under försöket analyserades dösarnas mineraloljehalter samt studerades temperatur och ett flertal mikrobiologiska variabler. Nedbrytningen av mineraloljor var snabbast under den första månaden. Detta avspeglades även i den höga mikrobaktiviteten under början av perioden. Den högsta uppmätta temperaturen i dösarna var 44 C.

Under en period av fem månader reducerades dösarnas mineraloljehalter från 2400 mg till 700 mg per kg mark (torrsubstans). De tillsatta mikroberna påverkade ej i hög grad nedbrytningen. I en av dösarna, till vilken icke tillsatts näringsämnen, var nedbrytningen till en början långsammare, men mineraloljehalterna sjönk senare till samma nivå som i de andra dösarna.

Kostnaderna för kompostering kan på basen av denna undersökning beräknas till i medeltal 100 mk/m3 förorenad mark. I denna summa ingår icke utgrävnings- och transportkostnader.

Sakord (nyckelord)
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EXECUTIVE SUMMARY

The Finnish Petroleum Federation (FPF) as the customer and Water and Environment Research Institute (WERI) as the consultant made in 1993 a one year contract to evaluate the suitability of bioremediation in order to evolve later a basic procedure (code of practice) to clean-up polluted soil at closed service stations. According to a prestudy, practical renovation was done by off-site composting. The goal of the field-scale study was to test how far lubrication oil can be composted during one summer season in Finland and find out whether microbial inoculants would enhance the degradation rate.

To locate oil contaminated soil, site surveys were carried out on three service stations in the Helsinki area. According to the operational history of the sites there was no information on uncontrolled leaks into the ground. Soil and groundwater sampling showed contaminated soil in the back yard area and near the places were the storage tanks and the gasoline pumps had been. Contaminants were lube oil, gasoline and diesel oil.

The contaminated soil (altogether ca. 800 $m^{3)}$ was excavated and transported to an asphalted composting area at the Metsä-Tuomela waste treatment station in the Nurmijärvi municipality (35 km north of Helsinki). The soils were sieved and composted in piles with bark and nutrients. pH was adjusted by lime. Two different commercial microbial inocula were also added into the piles. The piles were aerated by turning with a screw-type mixer at two to four weeks intervals. The piles were covered with tarpaulins and sampled for chemical and microbiological analyses after each mixing.

The result shows that there was a distinct decline in the mineral oil concentrations during the composting. Two thirds of the original oily material was degraded during 5 months. The pattern was about the same in piles with low and high starting concentrations. The degradation was highest during the first two months. Due to the lower ambient temperatures in the fall and the loss of the easily degradable material, the bioactivity was substantially lower during the later period.

Added nutrients did not prove to be vitally needed but they seem to be essential for a rapid process. This is a benefit in our climate. External microbes had no significant effect on the process. Tarpaulins were useful in preventing evaporation and packing of the piles during heavy rain fall.

70 % reduction was reached and final concentrations in oil contaminated soil were about 700 mg kg^{-1} dw. The clean-up result was good although it does not tell the real power of composting, because of the rather low initial oil hydrocarbon concentrations. The result anyhow indicates, that the pattern is about the same in piles with low and high starting concentrations. For more heavily contaminated soil of would however be useful to find out the efficiency of the short time composting in our climate to reach the strict concentration limits suggested for dumping of contaminated soils.

The actual cost of this experimental full scale composting is around 70-80 FIM/m^3. The real cost, i.e. the current price considering routine composting in the same kind of circumstances (ca. 1000 m^3 contaminated soil) with earthwork (excavation and transportation) would be of the order of ca. 250 FIM/m^3. This is also the expences estimated by other contracters in another contex. Composting can be regarded as fairly moderate price method for remediating oil contaminated soil.

CONTENT

1 FOREWORD

Soil contamination and the alteration of the use of the buildings and plots for other industrial or housing purposes can cause a need for soil cleaning activities at service stations. Changes in the use of the plots are partly due to the reducing of the fuel distribution network in Finland in the future. If the ownership of the land changes, the landholder, according to the new Waste Act, has to report in detail to the buyer if the soil is polluted and which kind of wastes possibly can have contaminated the soil.

Expenses for remediation of a site can be remarkable depending on the methods chosen for the clean-up. Therefore it is very important to be able to choose the right method for each particular case.

According to the studies and remediations done in several countries biological means are appropriate and cost effective to remove mineral oil hydrocarbons. However, their applicability in the Finnish climate, especially for the remediation of oil spills at service stations, has not been investigated.

The Finnish Petroleum Federation (FPF) as the customer and Water and Environment Research Institute (WERI) as the consultant made in 1993 a one year contract to evaluate the suitability of bioremediation in order to evolve a basic procedure (code of practice) to clean-up polluted soil at closed service stations. This procedure should serve as guidelines which oil companies, authorities and enterprises which offer remediation services could follow.

The project started with a literature survey in order to find out the state-of-the art in soil bioremediation. Based on the survey, the technique to be used in the practical studies both in the field and in the laboratory was then chosen. Composting on the one hand and *in situ* bioventing and ground water treatment on the other hand proved to be the rational choices for the field experiment. For practical reasons off-site composting was chosen and *in situ* treatment was left to be performed later on in another context. The aim of the final project was to create a code of practice for the remediation of oil hydrocarbon contaminated soils at Finnish service stations.

2 BIOREMEDIATION IN FINLAND AND OTHER COUNTRIES; COMPOSTING VERSUS OTHER MEANS

During the last decade notable effort in the highly industrialized Western countries has been devoted to the development of remedial action techniques for contaminated soils. Several types of remediation methods are now available: clean-up of excavated soil, *in situ* clean-up of soil and polluted groundwater without soil excavation, isolation or immobililization of contaminated sites and excavation of polluted soil followed by disposal on a controlled site. Most of the attention has been paid to the development of clean-up techniques for excavated soil but during past years more and more complete or partial *in situ* clean-up has also taken place.

Generally there is a wide variety of remedial action techniques available for cleaning up chemically contaminated sites. For every case comparative evaluations of the alternatives are essential to be carried out in order to select the most favorable and cost-effective one for implementation. For the remediation of contaminated sites, *ex situ* treatment in thermal and soil washing plants and solidification/stabilization are the most commonly used techniques today.

In Finland, the most common clean-up practices for oil wastes and oil contaminated soil have been incineration, landfarming and composting (biopiles). Futhermore, the less contaminated soils (oil less than 2 % by weight) have been deposited on dumping areas. Only a few cases of composting have been well followed-up and reported.

The two reported cases (Valo 1988, 1991 and Martikainen 1990) have been on composting of waste oil sludges. The cases were very much alike; wood based bulking agents (chippings and bark), nitrogen-rich nutrients (urea or mixed fertilizer) were used and rather tall piles were turned with a loader. The maximum initial mineral oil concentrations was ca. 30-100 g kg^{-1} dw. The portion of easily biodegradable saturated and aromatic compounds was 70-80 %.

The reduction of the mineral oil was in both cases about two thirds during two composting seasons (summers). The result is fairly good. The reduction percentage obviously expresses the real remedial potential of the method. Microbes had degraded the oil compounds which were readily available for them. The rest of the oil substances would take a substantially longer time to be degraded. These recalcitrant compounds include the largest oil hydrocarbon

molecules (e.g. asphaltenes). In bulk they are visually observed as 'oil bitumen'. They are very resistant to microbial action.

In addition to the Finnish experiments, there has been the same kind of pile composting in Norway (Halmø 1985). The treated material was waste oil collected from a shoreline. Degradation efficiency in the Norwegian composts was of the same magnitude as in Finland, being some 65 % during one summer. The Norwegian report pointed out that the pine bark they used had a good ability to sorb oil compounds.

A factor which might have improved the degradation results is more effective mixing of the piles during the process. Aeration of the composts concerned was done by turning the piles with wheel loaders. This type of mixing is not very efficient. There will always be unmixed phases left. This means that some of the pollutants very likely remain inaccessible to the microbes. This is the case especially with oily slurries and similar materials.

The advantage of good mixing is very well seen in drum composters. Effective mixing and aeration in an experimental (2 m^3 drum) composting (Valo and Salkinoja-Salonen 1988) resulted in 70-92 % clean-up for oil contaminated soil during 12-40 days when the original oil concentration varied from 100 to 400 g kg^{-1} dw. Even better results were obtained in a bigger scale (200 m^3) composter (Munckhof and Veul 1990) where pollutants were diesel oil, petrol and lube oil.

As an alternative to turning the composts, aeration of the piles can be arranged by forced ventilation through a piping system. In this kind of 'static' composting separate pipelines are usually used for watering. Composting with forced aeration without mixing has proven to work well with PAH (polyaromatic hydrocarbons) contaminated soils (Berg and Eggen 1991). Forced aeration can also be combined with pile tilling which makes the biodegradation even more effective, but on the other hand the maintenance is more complicated. The double system has been successful for diesel oil contaminated soil treatment (Lei *et al.* 1993).

3 PURPOSE OF THIS STUDY

The aim of the work was to find out how effective biodegradation of oil contaminated soil can be obtained during one summer in the rather cold climate conditions of Finland. Based on the best available information preparation of composts for biodegradation was chosen. Two commercial inoculants were

tested. The purpose was also to create a well documented case.

It was also considered to carry out forced aeration in this project but for practical reasons (too many parameters for one time) the fixed system experiments were deferred to be performed in another context. A vigorous mechanical mixing was chosen to guarantee that compostable compounds (i.e. waste oil) were available for biodegradation and to ensure a proper oxygen suply. The additives (nutrients, inocula etc.) would also be properly mixed with the compost matrix by this mixing.

4 MATERIALS AND METHODS

Site investigations were done by the Water and Environment Research Institute (WERI). The oil companies owning the sites provided their own data for the basis of the survey. WERI (/technical research office) conducted the practical geohydro-logical and physical analyses including drillings, samplings and analysis of the soil samples. Soil gas samples were analyzed at Tampereen Aluetyöterveyslaitos (Tampere, Finland).

Physical-chemical analyses of the soil, water and compost samples were mainly done by the WERI research laboratory. Novalab Oy (Karkkila, Finland) performed the nutrient analyses.

The initial purpose was to treat only the waste (lube) oil contaminated soil which was available in the design phase of the project. However during the preparations and the earthwork more polluted soil appeared. The contaminants were gasoline and diesel fuels. It was decided to treat these soils at the same time, but physically separated from the lube oil contaminated soils.

The site surveys and collection of the soils took place from April to early June 1993. The piling was finished and the first mixing was done 18th of June, 1993.

4.1 Site surveys

Site surveys were carried out to locate oil contaminated soil. Three service stations (A, B and C) were investigated. A and B were rather old service stations still in active operation. Station A, where most of the contaminated soil were dug up, had an unpaved place where self service oil changes had been done earlier. It also had old underground storage tanks which were at the same time replaced by the

marketing company. Soil layers consisted of earth filling (ca. 1.5 m) on the top and layers of sand, sandy silt and gravel above the bedrock which was at the depth of 3.5-4.5 m. Station C, where most of the diesel oil contaminated soil was, had been closed a few years ago. All constructions had been demolished and some of the building materials had been dumped in pits where the storage tanks were dug out. Soil matrix was mainly sand and gravel containing some humus, glay and a layer of coarse sand at the lowest excavation depth. The soil from station B (only ca. 15 m^3) included mainly diesel oil contaminated fill. According to the operational history of the three sites there was no information about product leaks into the ground.

At the first stage soil vapour studies were performed. The samples were taken at the points where oil was expected to be found according to maps and other information on possible spillage. Perforated soil gas tubings were installed at the depth of 0.5-1 m. At station C studies were done only after most of the soil was excavated and transported away. The concentration of volatile organic compounds (VOC) was measured with a portable photoionisation detector HNU Model HN-101 (HNU Systems Inc., MA, USA). From the tubes where high vapour content was measured, gas samples were vacuum pumped (AIRCHECK SAMPLER Model 224-PCXRT, SKC INC., USA) through activated carbon ampoulles (SKC, Inc., USA) for later laboratory analysis.

At some of the measuring points the volatile hydrocarbon content of the soil gas was very high. The highest contents were measured near the old storage tanks, but also high contents were found in places where the gasoline pumps had been. The highest concentration measured was 29 300 mg m^{-3} (as VOC).

The plan for soil and groundwater sampling at stations A and B was made according to the soil gas information and station history. The third station (C) was not sampled because of the excavations on the site. It was decided to transport the polluted soil to the treatment area. Most samples were taken from near the places were the storage tanks and the gasoline pumps had been. Samples were also taken in the direction of the groundwater flow. Control samples were also taken upstream.

The total petrol hydrocarbon concentrations in the soil were mostly below 2000 mg kg^{-1} dw. The highest concentration of mineral oil in the soil (7200 mg kg^{-1} dw) was found in the immediate surroundings of the storage tanks. Also high contcentrations were found near gasoline pumps and on the area where self service oil changes (station A) had been performed during a period of about 20 years (in 1950s and 1960s). Outside these areas oil concentrations were below 10-50 mg kg^{-1} dw.

At station A groundwater samples were taken from 14 different points at different depths. The highest content (3.3 mg l^{-1}) of oil was found near the gasoline storage tanks. The explanation of finding oil in the groundwater could be that there was a layer of sand under an apparent impermeable topsoil layer of sandy silt. The tank holes were dug through the impermeable layer. That was the reason why oil was also found deep under the groundwater level.

4.2 Pretreatment of the soil

The contaminated soil was excavated from the three described service stations in the Helsinki area. The majority of the soil, containing waste lube oil and gasoline was obtained from the station A. Altogether there were ca. 800 m^3 soil (1200 t) consisting of approximately equal volumes of lube oil, diesel and gasoline contaminated soils. Each type of soil was transported to the asphalted composting area at the Metsä-Tuomela waste treatment station in the Nurmijärvi municipality (35 km north of Helsinki) and treated separately throughout the process.

In order to avoid stones and other rough material causing mechanical problems during mixing, the soils were sieved with a screen shaker (65 mm grid). The reject (143 t) consisted of all kind of coarse landfill material (stones, lumps of concrete and asphalt etc.). It was deposited on the neighbouring dumping area.

4.3 Piling

The sieved soils were placed with the bulking agent in separate piles (at first in 11 piles) of ca. 1.5-1.7 m high and 3.5 m wide. The length varied, being shortest, ca. 15-20 m in the five oil contaminated soil piles. In the beginning two diesel oil and four gasoline contaminated soil piles were constructed. They were reorganized (moved and combined) during the composting. At the end of the process there were one diesel oil and two gasoline contaminated soil piles. The ambient temperature during the preparing work was 15-20 °C.

The bulking agent was spruce bark. The total amount of bark used in the piles was approx. 300 m^3. In the lube oil and diesel oil contaminated soil piles the ratio of bark to soil was approx. 1:3 on a volume basis. In the gasoline soil piles the ratio was approx. 1:4. Piling was done layer by layer starting with bark. A voluminous layer of bark (thickness approx. 25 cm) was placed at the bottom to ensure good venting and maintenance of aerobiosis in the piles. Also the bark served to absorb leachable oil compounds. Altogether there were three layers of both soil and bark.

Table 1. Nutrient and inocula amendments in the lube oil contaminated soil piles

Pile no	Nutrients	Inocula
1	+	X
2	+	-
3	+	Y
4 (control)	-	-
5	++	-

4.4 Nutrient amendments

Nutrients were added as dry nitrogen rich compound fertilizer (Typpirikas Y-lannos 1, Kemira, Finland). The product contained 26 % N, 3 % P, 3 % K, 1.5 % S, 0.5 % Mg, 0.03 % B and 0.0006 % Se. Nitrogen compounds consisted of NH_4-N (54 %) and NO_3-N (46 %). The dosing of the fertilizer was done according to the oil hydrocarbon content of the soil. In the five waste oil contaminated soil piles the practical addition was roughly 1 kg fertilizer per cubic meter compost matrix. In the control pile no nutrients were added. In one of the piles the dosage was initially about double the average. Later on (60 d and 95 d mixing) half of the original dosage was used. For the rest of the piles (containing diesel oil and gasoline) with lower concentrations of pollutants dosing was less. Nutrients were added manually on the top of the piles before the mixing.

4.5 pH adjustment

The original pH of the contaminated soils varied in the range of 6.0 - 6.5. The pH of the bulking agent (spruce bark) was 4.9. The pH of the composts was adjusted by fine granular lime (Kalkki 2, Nordkalk Oy, Sipoo, Finland). The addition was done according to the measured pH values and the known buffering capacity of the contaminated soil types. The target value was neutral or slightly basic. Adjustment resulted in starting pH values of 7.1 - 7.4 in the lube oil and diesel oil contaminated soil piles and 7.6 - 7.9 in the gasoline soil piles.

4.6 Inocula

For the composting of lube oil contaminated soil two commercial inocula were used. One was a dry powder consisting of a mixed culture of oil degrading

bacteria, trade name 'PRC 107 DTX' (Interbio Inc., USA, distributed in Finland by Biocora Oy) and the other one, 'Oilbac' (Ecolution Oy, Helsinki, Finland) was a matrix of surface soil from a landfarming site used for the disposal of oily wastes. The inocula were added manually on the top of the piles just before the first mixing.

4.7 Mixing and aeration of the piles

The piles were aerated by turning with a wheel loader drawn or tractor drawn screw-type mixer (Anon., RTA Virtanen Oy, Hyvinkää, Finland and Allu 24, Ideachip Oy, Hollola, Finland). Turning was first (June-July 1993) done twice a month. Later on the piles were turned only once a month. The last mixing was done in September 1993.

After mixing the piles were covered with tarpaulins. Covering was to prevent drying and wetting of the piles and also to diminish the release of volatile compounds from the system. A minor part of the piles was left uncovered. This was to test how much the piles were affected when exposed to changeable weather conditions. The total volume of the completed piles was about 750 m^3 (i.e. 800 m^3 soil minus material rejected by sieving plus 300 m^3 bark compressed when mixed with heavier soil).

4.8 Sampling

Compost samples for chemical and microbiological analyses were taken every time the piles were turned (mixed) for aeration (see above). Each pile was sampled from 10-12 random locations at 0-20 cm depth. Because of the vigorous mixing of the piles prior to sampling it was considered unnecessary to sample at lower depths. The composite samples (approx. 15-20 l per pile) were sieved (8 mm grid), collected in glass jars and stored at +2 °C if not analysed immediately. On one occasion (November 1993) the composite sample was taken in duplicate, to see the variance between the samples.

In order to monitor volatile degradation products in the piles and to estimate the loss of oil hydrocarbons into the atmosphere compost gases were also analysed. The samples were taken at about the same interval as the others but after mixing the piles were left to 'equilibrate' one to two days before sampling. Gases were vacuum pumped (Aircheck Sampler Model 224-PCXRT, SKC Inc., USA) from perforated tubings (1-2 pcs per pile) which were set at approx. one meter depth in the piles. Volatile hydrocarbons were absorbed onto activated carbon ampoulles

(SKC Inc., USA) for later analysis. CO_2 and moisture samples were taken by the Dräger Gas Detector system (Dräger, Drägerwerk ag Lübeck, Germany).

4.9 Microbial respiration in the soil compost piles

The objective of the laboratory study was to assay the bioactivity of the pile matrices during the course of the composting. This was done by determining the basic soil respiration (actual microbial activity) and substrate induced respiration (potential microbial activity) according to the German Dechema laboratory methods (Dechema 1992) with some modifications. In short, field moist soil corresponding to 10 g dry soil was incubated at 22 °C in 126 ml glass flasks sealed with teflon lined rubber stoppers. The evolved CO_2 in the headspace was then measured. For the basic respiration the bottles were incubated up to two days. For the substrate induced respiration samples were incubated for two hours. This short incubation time does not allow time for exponential microbial growth, but according to Anderson and Domsch (1978) this immediate activity is a direct measure of the microbial biomass size. In the substrate induced respiration carbon, nitrogen and phosphorus were added as follows: 100 mg of glucose, 15 mg of NH_4Cl and 2 mg of KH_2PO_4 per 10 g dry soil.

4.10 Enumeration of bacteria

The number of colony forming heterotrophic bacteria in compost samples was determined by plating a dilution series on tryptone-glucose-yeast extract medium (TGY) diluted to 1:5 (American Public Health Association, 1989). Also enumeration of hydrocarbon degrading bacteria was attempted on a mineral medium containing motor oil, diesel or gasoline as the sole carbon source. The mineral medium contained (in milligrams per liter) the following: KH_2PO_4, 8.5; K_2HPO_4, 21.7; $Na_2HPO_4 \cdot 2H_2O$, 33.4, NH_4Cl, 0.5; $CaCl_2 \cdot 2H_2O$, 39.4; $MgSO_4 \cdot 7H_2O$, 22.5; $FeCl_3 \cdot 6H_2O$, 0.2; cycloheximidine, 150; noble agar, 1500. Different methods for addition of motor oil were tested: sonication of hot agar solution after autoclaving, application of hexane-acetone solubilized oil to solidified plates (200 µl of oil per plate (15-20 ml of medium). Gasoline and diesel (200 µl) were added to a small piece of filter paper which was placed in the lid of the petri dish. These plates were incubated in a closed glass jar. The final amount of the carbon source available in the solidified medium was approximately 1 % (w/w). Control plates were incubated without any carbon source.

4.11 Isolation and identification of bacteria from inocula and compost piles

Colonies were isolated from TGY plates with the highest dilutions. These were anticipated to represent the most numerous culturable bacteria. Approximately 10 colonies were isolated from each of the following samples: inoculum PRC 107 DTX, inoculum Oilbac, original lube oil contaminated soil, compost pile 2 (no inoculum) and compost pile 3 (inoculated with PCR 107 DTX).

The isolates were purified by restreaking single colonies three times on TGY (1:5) solid medium. The identification of isolates was done by API (bioMerieux sa, France) and Biolog (Biolog, Inc., Ca., USA) identification systems. [The API 20 NE (non enterobacter) system is based on 9 different physiological traits and the assimilation of 12 different carbon sources. The data base contains 66 species. The API 20 E (for enterobacteria) is based on 12 physiological tests and the fermentation of 9 different sugars. The data base contains 108 species. The Biolog system is based on the assimilation of 95 different carbon sources. The database for gram negative bacteria contains 569 species and for gram positive bacteria 225 species.]

4.12 Denitrification in soil compost piles

Denitrification is the reduction of nitrate (NO_3^-) to gaseous nitrogen (N_2) during the oxidation of organic matter by bacteria (Tiedje, 1988). In other words nitrate is used as an alternative electron acceptor instead of oxygen in bacterial respiration. Denitrification only occurs when the oxygen concentration is very low or in the absence of oxygen. Denitrification affects the microbial activity in two different ways: (i) an efficient respiration can continue in the compost piles if oxygen is exhausted and hydrocarbons and their degradation products may be degraded, (ii) the nitrate converted to nitrogen gas is a loss of nitrogen source for growth.

Denitrification in the compost piles was followed during the course of the composting season. The rate of denitrification was measured using the acetylene blockage method (Tiedje, 1982). In this method the accumulation of the intermediate nitrous oxide (N_2O) measured in the presence of acetylene (C_2H_2), which inhibits the further reduction to N_2.

Natural moist soil samples corresponding to 10 g of dry soil were incubated in 126 ml flasks sealed with teflon lined rubber stoppers. Half of the bottles were flushed

with nitrogen, N_2 to create anaerobic conditions before the addition of acetylene. Acetylene was added with a syringe through the rubber stopper to a final concentration of 9 % (vol).

The flasks were incubated at 22 °C in the dark. Each sample was prepared in triplicate. The accumulation of N_2O was measured by withdrawing a 0.5 ml gas sample and injecting it into a gas chromatograph equipped with an electron capture detector. Separation was done on a 1.8 m long porapak Q column using a carrier gas flow of 30 ml min^{-1} ($ArCH_4$(5 %)). Samples with atmospheric composition in the headspace were incubated for up to one day and samples with a nitrogen atmosphere were incubated up to eight hours.

4.13 Chemical and physical analyses

Compost matrices were analyzed for the total oil hydrocarbon (oil and grease) content (i.e. compounds soluble in carbontetrachloride) and the mineral oil content with an in house method based on Finnish standard SFS 3010. The procedure includes an extraction of the field moist or air dried samples with CCl_4 in an ultrasonic bath and measurement of the filtered extract by infrared spectrometer. The results for total oil hydrocarbons and for the mineral oil content are obtained before and after the removal of polar material by aluminium oxide column separation, respectively.

To determine the hydrocarbon composition of the samples, the filtered extracts were also analysed by GC-FID. This was performed on samples of the original contaminated soil, bark chips and the compost mixture during the composting time. A gas chromatograph (HP 5890 A) fitted with a SE-54 columnn was used. A splitless injection of 1 µl was used and the oven program was as follows: 40 °C for 4 mins, ramp from 40 °C to 280 °C at 10 °C/min and 280 °C for 32 mins. The chromatograms were examined only visually. Quantitation or fractionation on the silica gel column into aliphatic and aromatic components was not performed at this stage.

CO_2 analyses for the soil respiration tests were conducted by EASY QUANT EQ 90 analyzer (Easy Quant Ay, Lammi, Finland). The method involves headspace analysis with IR spectrometry. CO_2 and H_2O measurements for the compost gases were done by the Dräger Gas Detector system (Dräger, Drägerwerk ag Lübeck, Germany).

Nutrients (N, P) were analyzed by the Kjeldahl method (nitrogen) and plasma

emission spectrophotometry (phosphorus). NH_4-N and NO_3-N assay included sample extraction by 0,1 M K_2SO_4 followed by distillation (NO_3-N with Devarda reagent) and titrimetric analysis. The nutrient as well as pH measurements were done by Novalab Oy (Karkkila, Finland).

Soil moisture was measured by drying (at 105 °C) to constant weight.

Temperature measurements in the composts were done at three to five locations per pile at ca. 50 cm depth. The pile temperatures recorded are the averages of the measured values. The thermometer (Testo 700, Testoterm GmbH & Co, Lenzkirch, Germany) included a measuring instrument and a rigid probe up to 3 meters long.

5 RESULTS

5.1 Chemical composition of the sieved contaminated soil and bulking agent

The chemical composition of the sieved soils and bulking agent before preparation of windrows is given in Table 2. The classification of the contaminated soil types as waste oil, diesel and gasoline contaminated soil was based on the analyses done during the site investigation. Note that only total hydrocarbons were measured (not mineral oil).

Table 2. Chemical analysis of sieved contaminated soil and bulking agent

Matrix	Total hydrocarbons mg kg^{-1}	N_{tot} mg kg^{-1}	NH_4^+-N mg kg^{-1}	P_{tot} mg kg^{-1}	pH	Dry matter %	Org. matter %
Waste oil contam. soil	2600	780	23	310	6.6	87	3.6
Diesel contam. soil	1600	1490	54	284	6.0	76	11.4
Gasoline contam. soil	500	300	22	220	6.2	91	3.5
Bulking agent	15000	4000	90	310	4.9	41	97
Oil bac (inocula)	20200	ND	ND	ND	ND	ND	ND

ND: not determined

5.2 Oil hydrocarbon degradation

Monitoring of the mineral oil was first done after the initial mixing of the piles. Follow-up monitoring was repeated every time mixing took place. The only

exception was the last sampling in November when no mixing was performed. The results for the composting of the three different kinds of oil hydrocarbon contaminated soils are shown in Figs 1 and 2. The degradation in the piles containing lubricating oils are shown in Fig. 1. Diesel oil and gasoline pile values are shown in the Fig. 2. The latter ones have been calculated as averages of concentrations found in the same sort of piles. (Note the reorganizing of the diesel oil and gasoline piles during the composting process.)

The presented data is obtained from extraction of field moist samples. Control experiments with air dried samples during the last two samplings showed that the mineral oil content in the field moist samples was underestimated up to 26-73 % depending on the concentration level. The relative standard deviation for the mineral oil content of the duplicate composite (n = 7) samples (last sampling in November 1993) was less than 30 % (4 -30 %) in the field moist samples and less than 14 % (0 -14 %) in the air dried samples.

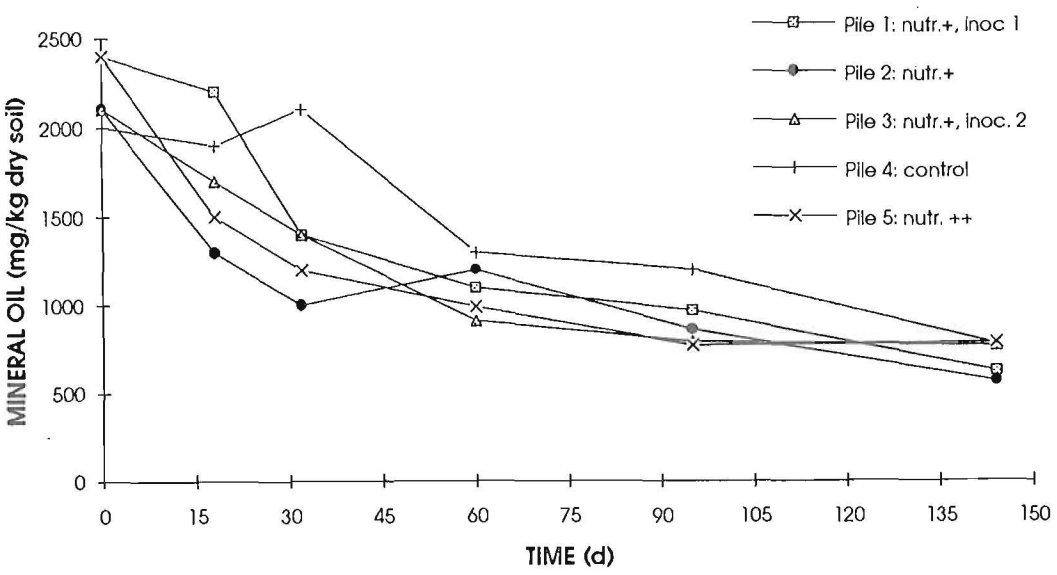

Fig. 1 Degradation of mineral oil in lube oil contaminated soil piles.

There is some variation (approx. 300 mg kg^{-1}) in the initial concentrations (time 0) of the parallel five lube oil piles. That is probably due to the heterogenous nature of the piles. On the other hand the 'Oilbac' inocula (pile nr 1) certainly contained some oil hydrocarbons which are likely to contribute to the total concentration. Otherwise the pattern of the decline is very clear. The degradation was most rapid during the first month. The rate declined with time. Lube oil compounds were degraded at the same rate in all piles but the control pile (4), which also reached the same rate after starting.

The residual concentrations after five months composting were roughly one third of the original. All the different sort of piles with 2600, 1600 and 500 mg total hydrocarbon per kg dw starting concentrations had same magnitude of reduction. This was the same in all the piles irrespective of the type and level of contamination.

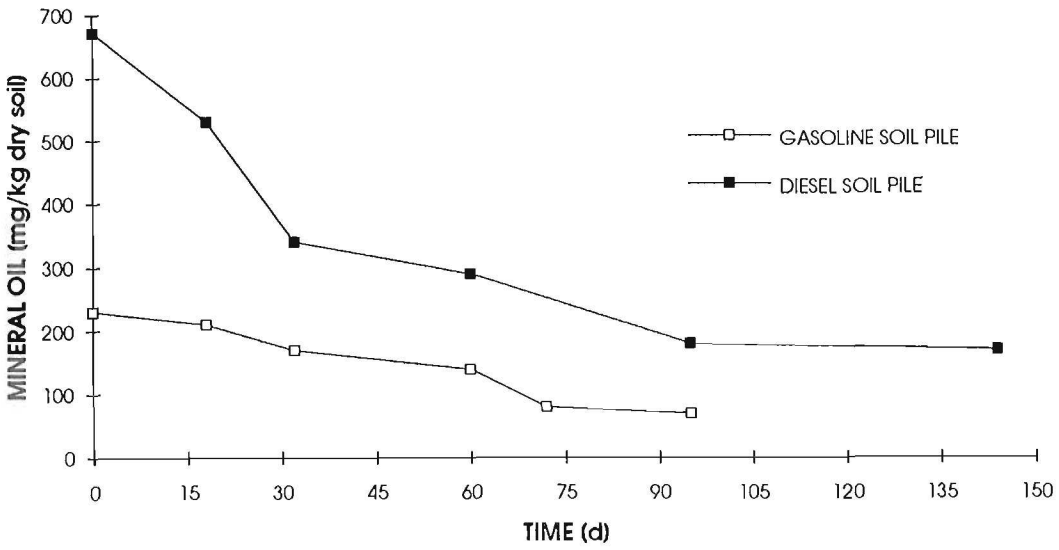

Fig. 2 Degradation of mineral oil in diesel and gasoline contaminated soil piles.

The degradation of gasoline and diesel oil follows the same pattern as lube oils. Only the relative amounts are smaller. The figure also shows that the concentration of gasoline is already quite low after sieving prior to composting. Part of the typical compounds of the gasoline and diesel had apparently volatilized during the excavation and sieving of soil even though they were found during the site investigation.

The contamination in the original soils did not resemble the pure oils (lube oil, diesel and gasoline) even though they were classified in this way (Figs. 3 A and B). For instance the so called gasoline contaminated soil did not include any characteristic signals for gasoline even if the moist soil was extracted. Also the lube oil and diesel resembled each other as if they were contaminated with diesel and lube oil. In the piles containing so called diesel contaminated soil the amount of diesel compounds was higher than in lube oil contaminated soil piles. The chromatograms of lube oil and gasoline contaminated soil piles looked the same. Only the concentration was higher in lube oil piles.

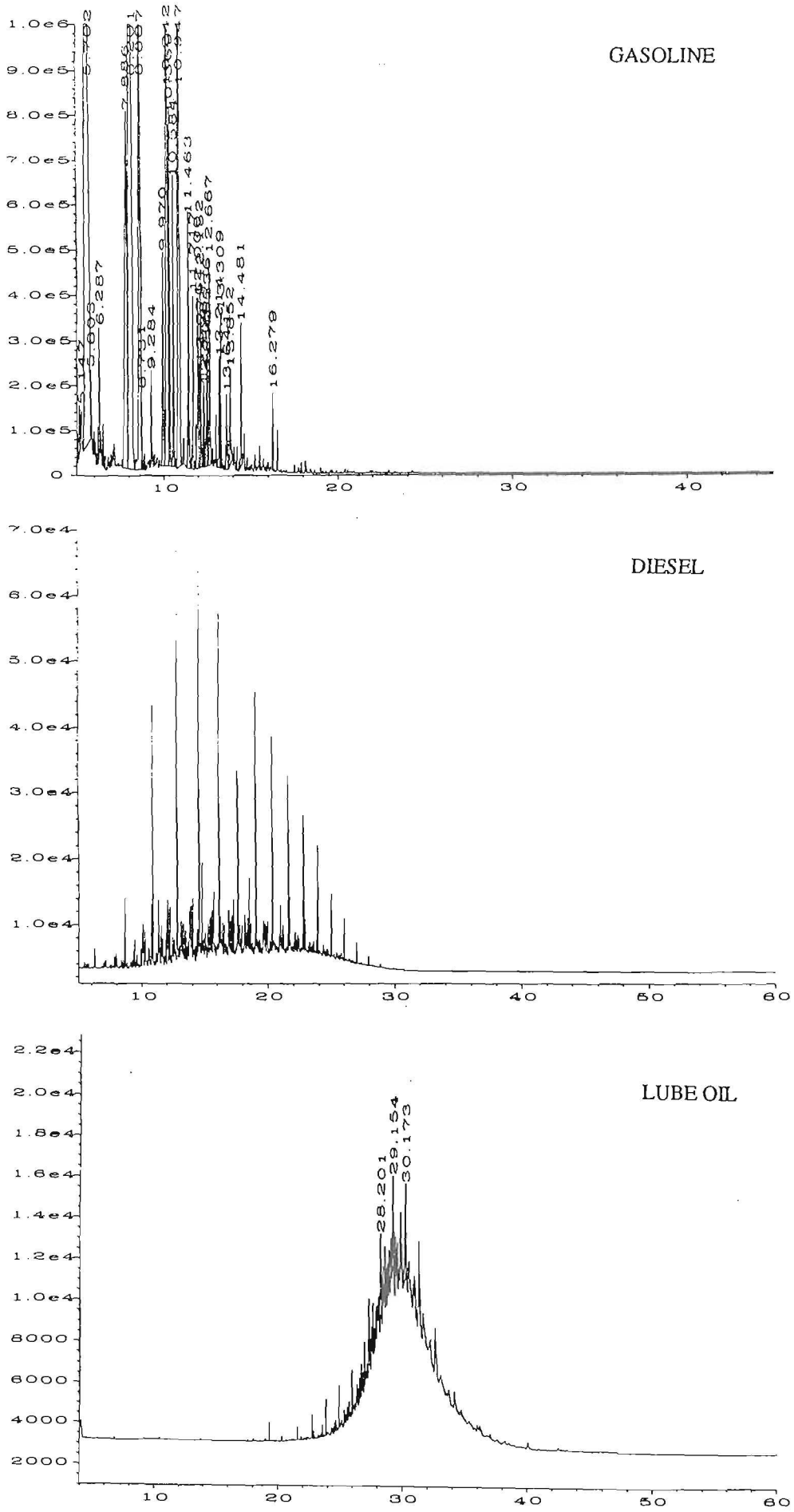

Fig. 3 A Chromatograms (GC-FID) of CCl_4 extracts of pure oil products

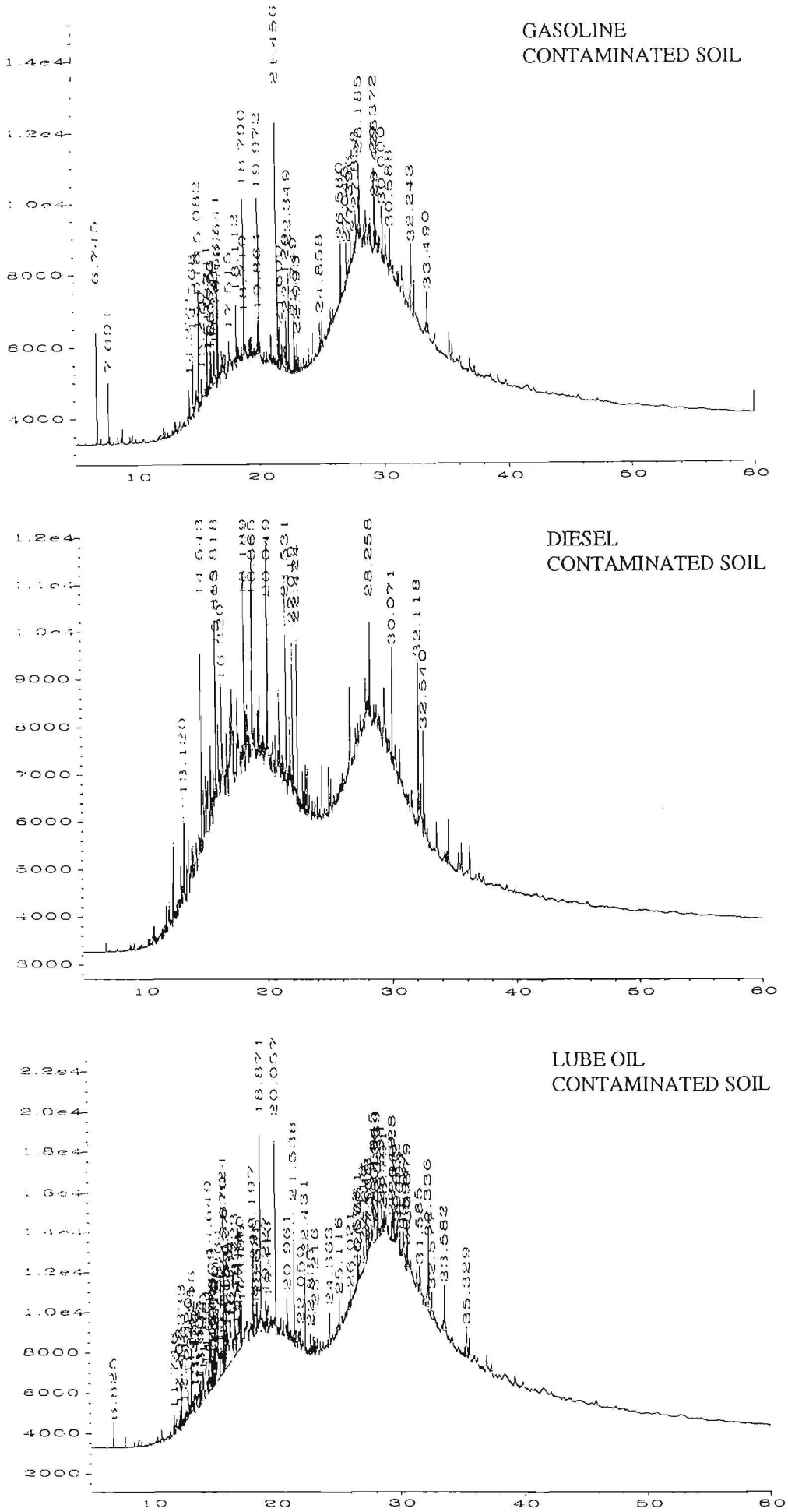

Fig. 3 B Chromatograms (GC-FID) of mineral oil content of lube oil, diesel oil and gasoline contaminated soil compost matrices (time 0).

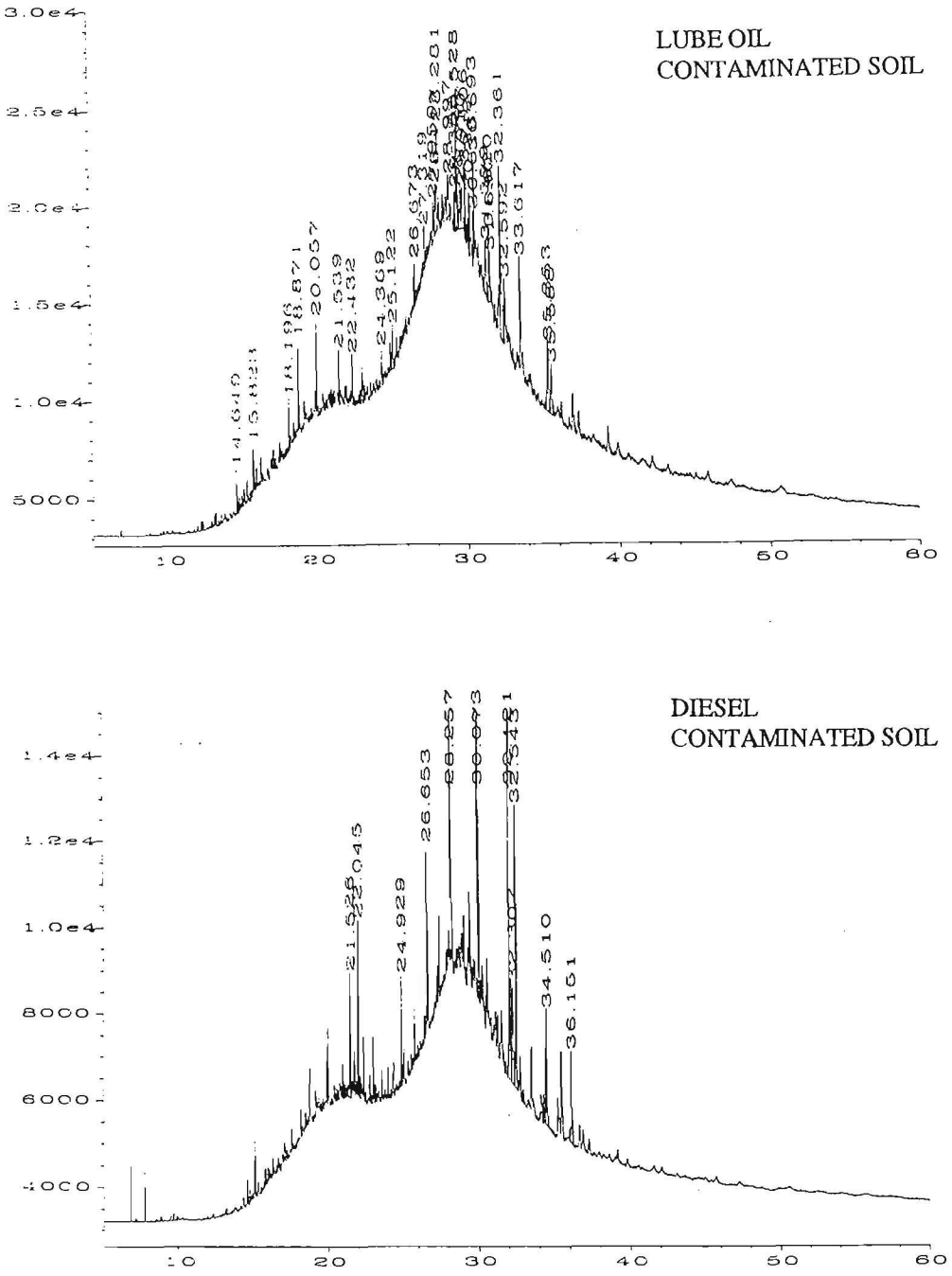

Fig. 4 Chromatograms (GC-FID) of mineral oil content of lube oil and diesel oil contaminated soil compost matrices (time 5 months).

Bark chips contributed extra signals to the chromatograms. They disappeared however for the most part in one month. They were not seen in the compost matrice extracts purified with Al_2O_3 column before GC run.

After five months the GC trace of the lube oil compost matrix resembled the original soil although there were a significant reduction in the unresolved 'envelope'. Also many of the resolved peaks (not alkanes) had decreased.

For the diesel and gasoline compost matrices there was not much change during the processing. The concentrations of the extracts were however too low for the GC-FID since they were not concentrated prior to analysis. The residual gasoline seems to have degraded similarly to lube oil. In the diesel oil contaminated soil piles, which also contained compounds typical for lube oil, has diesel part degraded mostly.

5.3 Composting temperatures

The summary of the lube oil pile temperatures is in Fig. 5.

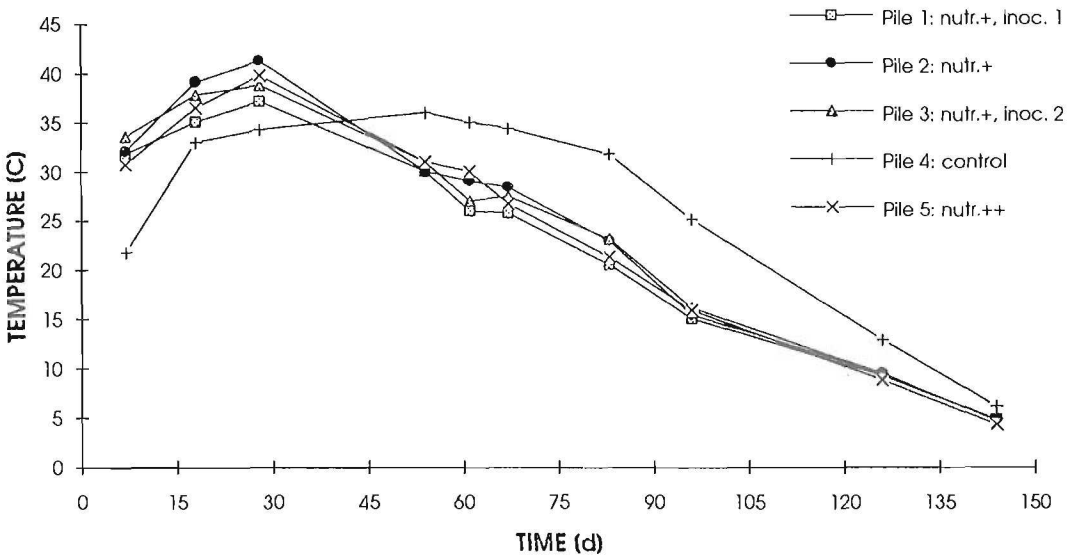

Fig. 5 Temperatures of lube oil contaminated soil piles

Fig. 5 clearly shows a distinct increase of pile temperature during the first month of composting. Only in the control pile was the temperature rise somewhat slower, reaching the maximum after two months of processing. The control pile also remained remarkably warmer for about 30 days longer. The maximum temperatures were about the same for all of the piles the highest slightly exceeding 40 °C. The latest measurements were done in November. At that time the outdoor temperature was -2 °C and the compost temperatures had fallen to around +5 °C. During September, the ambient temperature had already been near zero and during the nights was even lower (-5 °C).

5.4 Volatile hydrocarbons in the compost gases

The gas phases in the piles were analysed for total volatile organic compounds (TVOC). The summary of the measurements (Fig. 6) shows that volatilisation of the oil hydrocarbons happened during the first few weeks of processing. It stopped in one and a half months time.

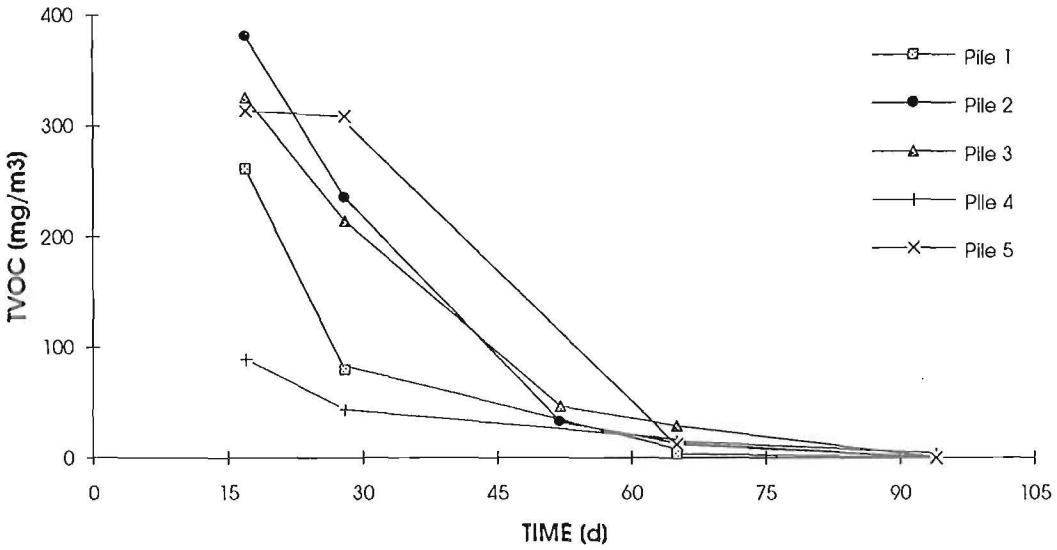

Fig. 6 Total volatile organic compounds (TVOC) in lube oil contaminated soil pile gases

Volatile losses were lowest in the control pile (4). In contrast to all the other piles it is only one fourth or fifth of the quantity.

5.5 Microbial respiration

Biodegradation activity in the piles was monitored by soil respiration (CO_2 production) tests and bacteria enumeration. Basic soil respiration which reflects the actual degradation of organic matter (hydrocarbons and other organic matter) is summarized in Fig. 7.

Respiration in the lube oil piles seems to follow the same pattern as the temperatures do. The control pile peaks later but the activity is higher than in the others after two months composting.

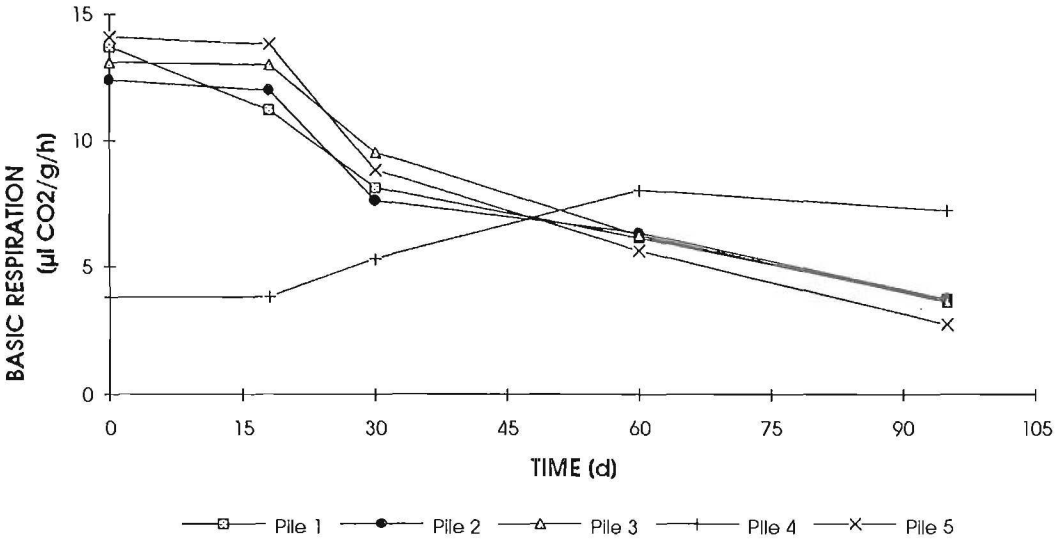

Fig. 7 Basic respiration in lube oil contaminated soil piles

The results of the substrate induced respiration (so called potential bioactivity) measurements, where glucose, N and P were added to all sample bottles, are summarized in Fig. 8.

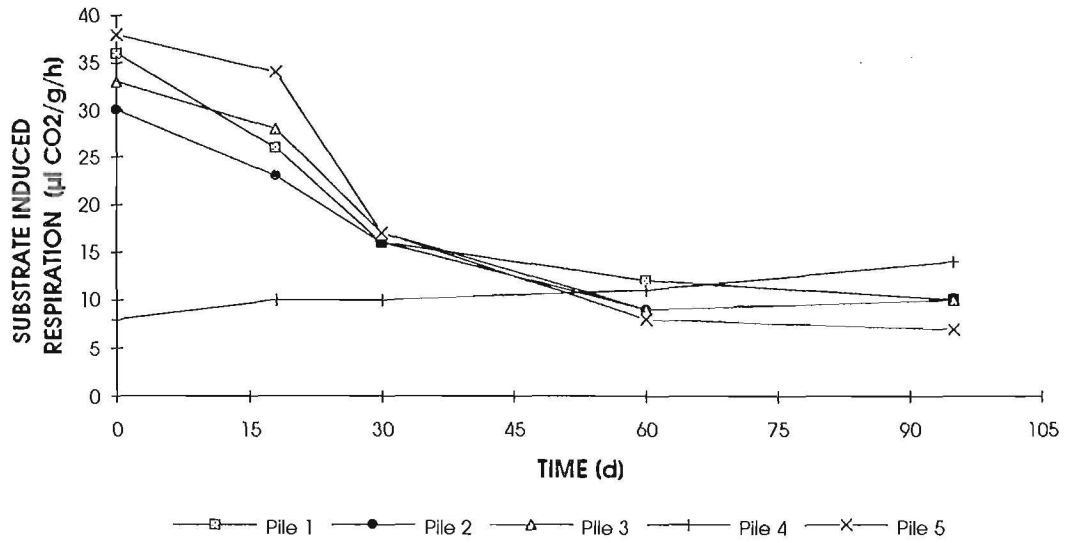

Fig. 8 Substrate induced respiration in lube oil contaminated soil piles

Substrate induced respiration (SIR) in the lube oil piles shows the same chronological pattern as the endogenous respiration. Substrate induced respiration was about three times higher than endogenous respiration. A distinct exception is the control pile SIR activity. It increased slowly during the season as did the basic (endogenous) respiration.

5.6 Enumeration of bacteria

The numbers of culturable heterotrophic bacteria in the lube oil compost piles are given in Table 3. The counts did not change significantly during the composting period. The number of bacteria in the sieved soil before the preparation of the piles was $1.8 \cdot 10^7$ CFU g^{-1} (dry soil). The number increased about 10 fold immediately after the preparation of the compost piles. The heterotroph counts from diesel and gasoline contaminated soil composts ranged from $7.5 \cdot 10^7$ to $6.8 \cdot 10^8$ CFU g^{-1} (dry soil).

The plate counts of oil degrading bacteria did not succeed very well. The addition of lubricating oil to the mineral medium created an irregular surface and the counts were unreliable. Plates incubated with diesel or gasoline as the carbon source in the headspace were possible to count. The control plates without any carbon source,

Table 3. Number of heterotrophic bacteria in compost matrices of lube oil contaminated soil. Determination by spread-plating on TGY (1:5) solid medium.

Pile	Colony forming units [10^8 CFU g^{-1} (dry soil)]				
	Days of composting				
	0 d	18 d	32 d	60 d	95 d
1	4.6	5.1	8.2	0.8	3.6
2	3.5	1.7	1.6	1.5	2.3
3	2.6	2.6	ND*	2.2	2.9
4	ND	4.0	ND	1.9	4.4
5	ND	7.7	5.5	1.5	4.4

* Not determined

however, gave about the same count or even higher than the ones incubated in the presence of diesel or gasoline. That might be be due to inhibition by gasoline or diesel. Counts on mineral medium ranged from $3.0 \cdot 10^7$ to $1.8 \cdot 10^8$ CFU g^{-1} (dry soil) and mineral medium plus gasoline or diesel as the carbon source counts ranges from $5.3 \cdot 10^7$ to $1.5 \cdot 10^8$ CFU g^{-1} (dry soil). There was no distinct pattern in the counts (data not shown).

5.7 Identification of bacteria isolated from inocula and compost matrix

Four different bacterial species were identified in the PRC 107 DTX inoculum: *Enterobacter sakazakii*, *Bacillus mycoides*, *Klebsiella oxytaca* and *Acinetobacter calcoaceticus*/Gen 13. In the Oilbac inoculum bacteria were identified as *Bacillus megaterium*, *Pseudomonas diminuta*, *Gluconobacter cerenius*, *Pasteurella caballi* and one species beloning to the genus CDC plus several unidentifiable species.

In pile 2 which was not inoculated we found *Pseudomonas paucimobilis*, *Sphingobacterium multivorum* and several other bacteria that did not match any of the data bases of the identification systems. In pile 3, which was inoculated with PRC 107 DTX, we identified isolates as *Pseudomonas vesicularis*, *Sphingomonas paucimobilis*, *Sphingobacterium mizutaii*, *Sphingobacterium multivorum* and CDC group DF-3 and several unidentifiable bacteria.

None of the species present in the PRC 107 DTX inoculum were found among the ten colonies isolated from the most dilute plate of the inoculated compost windrow (3).

5.8 Denitrification

The rates of denitrification in the compost matrices are shown in Fig. 9 (aerobic conditions) and Fig. 10 (anaerobic conditions). The rates under aerobic conditions are ranging from 0 to 18 ng N_2O-N g^{-1} (dry soil) h^{-1} and under anaerobic conditions from 0 to 2285 ng N_2O-N g^{-1} (dry soil) h^{-1}. The rate under anaerobic conditions is thus more than 100 times higher. The real oxygenation state in the compost piles is probably in between these two limits as is the actual denitrification rate. Under both aerobic and anaerobic conditions the rate of denitrification decreased during the composting time.

Nutrient analyses were performed mainly in order to see which kind of correlation can be seen between degradation rates versus total and soluble nutrients (N, P). Approximately 1 kg of fertilizer (Typpirikas Y-lannos) was added per m^3 of compost matrix. One m^3 of compost weighs about 1200 kg and has a dry matter content of about 85 %. This means that 1000 g fertilizer per 1200 kg compost (fresh weight) = 0.83 g fertilizer per 1 kg compost or 0.98 g fertilizer per 1 kg compost (dry weight) was added. The fertilizer contained 26 % N distributed with 54 % ammonium N and 46 % nitrate N. According to this calculation about 138

mg of NH_4^+-N and 117 mg of NO_3^--N was added per kilo of compost (dry matter).

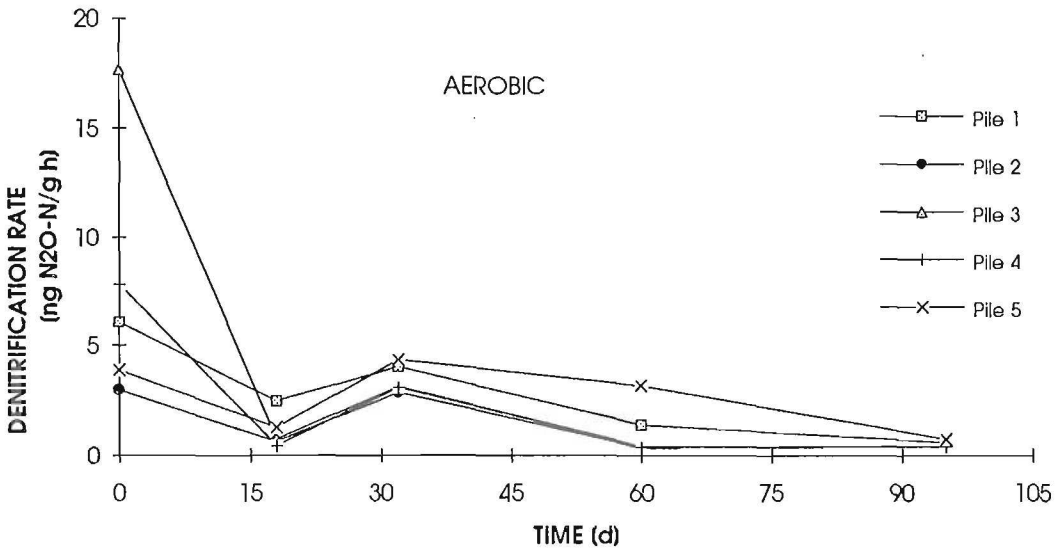

Fig. 9 Rate of denitrification in lube oil contaminated soil pile matrices (aerobic conditions)

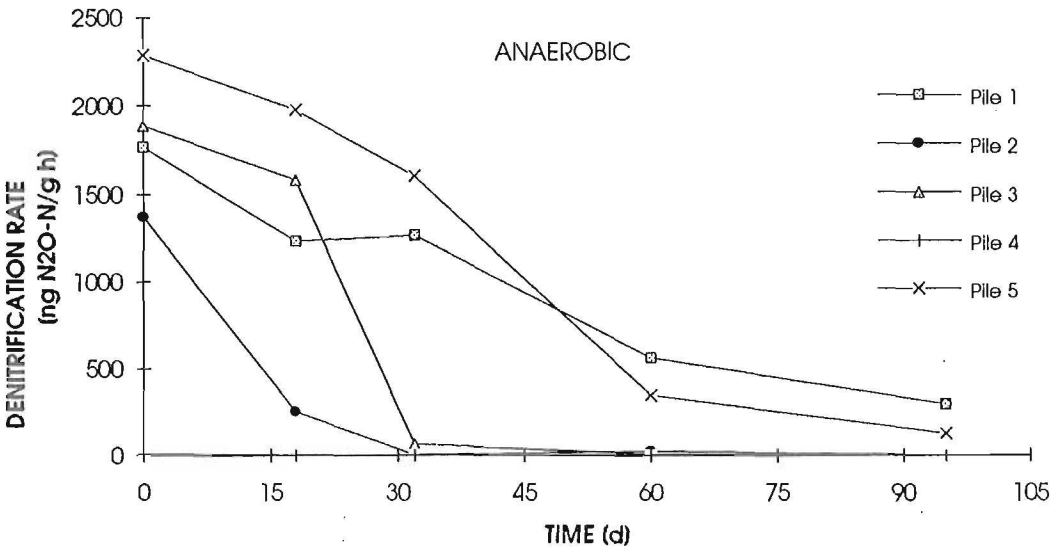

Fig. 10 Rate of denitrification in lube oil contaminated soil pile matrices (anaerobic conditions)

The measurements of soluble nutrients (NH_4^+-N and NO_3^--N) were done only at the samplings after 60 and 95 days (Table 4). The soluble nutrients addition to pile 1, 2, 3 and 5 apparently enhanced the microbial activity during this phase and

after 60 days the soluble nutrient levels had declined to the same level as in pile 4.

The pile (nr 5) which received a double dose of the fertilizer did not show any higher degradation even though the soluble N ($NH_4^+$and NO_3^-) were available in abundance (Fig. 7).

Table 4. Concentrations of nutrients in the five lube oil piles during composting.

Date	Days	Pile no	N_{tot}	NH_4^+-N	NO_3^--N	P_{tot}
			mg kg^{-1} (dry weight)			
6.7.93	32	1	1200	ND*	ND	610
		2	1120	"	"	390
		3	1230	"	"	370
		4	1060	"	"	320
		5	1130	"	"	350
19.8.93	60	1	1180	2.6	15	475
		2	1290	2.6	5.3	480
		3	1450	5.4	2.7	505
		4	940	5.3	2.6	415
		5	1520	223	141	550
23.9.93	95	1	1290	8.3	<0.1	480
		2	1210	8.2	<0.1	415
		3	1320	11	2.5	405
		4	1150	8.2	5.2	365
		5	1650	128	287	590

* Not determined

6 DISCUSSION

6.1 Oil hydrocarbon analyses

Mineral oil hydrocarbon analyses for soils were performed by in house IR-method. Measurements were done on field moist samples. Air drying of the samples before extraction was avoided because of the loss of volatile compounds.

Drying can, however, be done chemically by using sodium sulphate (Na_2SO_4) and this appears to be important when using a non-polar solvent for extraction. Sodium sulphate absorbs water which interferes the extraction of oil hydrocarbon compounds. The yield can thus be improved. This was learned in the course of the study.

The analytical data presented here are therefore to some extend underestimated. However, they are sufficient to follow the trend of degradation and the efficiency of biodegradation and to make other conclusions. But they are also an implication for the future work to put much effort on adequate analytical methods.

In general, much attention is nowadays paid to the methods for analyzing oil hydrocarbons in soils. It has been noticed that, depending on the soil matrix and the quality of the pollutant one often needs different kind of methods for the same sample to achieve a qualitatively and quantitatively reliable data.

6.2 Biodegradation of oil hydrocarbons

The results of the experimental piles show that there is a distinct decline in the mineral oil concentrations during the composting. Two thirds of the original oily material was degraded. The maximum degradation intensity was highest during the first two months. Due to the lower ambient temperatures in the fall and the loss of the easily degradable material (seen also by eye) bioactivity was substantially less during the later period.

There seems to be about the same degradation pattern for the hydrocarbon residues left in the originally waste oil, gasoline and diesel contaminated soils. The result also indicates that the rate is about the same in piles with low and high starting concentrations. This has been pointed out also by other research groups (ref. Shell Research Center 1994) even though the initial concentration were in the range 10 000 - 50 000 mg kg^{-1} dw.

As judged from the gas chromatograms (Fig. 3) the hydrocarbons present in the soil not resemble the chromatograms of fresh products. Apparently evaporation of volatile compounds have taken place during the initial digging out, transport and sieving of the soil. The hydrocarbon residues left in the soil seemed however to degrade during the composting. Due to the health and environmental risks associated with the loss of volatile compounds, composting of gasoline contaminated soil should not be recommended.

The degradation of hydrocarbons is mainly thought to be an aerobic process, at

least the initial oxidation of the terminal end methyl group. Degradation products of hydrocarbons and monoaromatic hydrocarbons (BTEX) can also be degraded under anaerobic conditions in the presence of nitrate through the denitrification process (e.g. Schocher *et al* 1991). The rather frequent turning of the piles to ensure good aeration was probably one reason for the good result.

The microbial respiration measurements correlated with the degradation pattern of mineral oil. The bacterial plate counts did not follow any particular pattern and respiration may be a more useful tool to detect microbial activity in this type of environment.

6.3 Nutrients

The soils treated were rather poor in readily available nutrients. This is often the case with mineral soils. Although soils have reasonable reserves of organic N (see p. 13, Table 2), the main nutrient usually needed is nitrogen (ammonium). The level of contamination may also have meaning wheather the system is N limited or not.

There are several ways to amend nutrients in the composting. Instead of artificial fertilizers one can also use e.g. waste water sludges. The best control is however gained with compound fertilizers. In addition they also include trace nutrients which may enhance the composting.

It was expected that without any nutrient addition the microbial activity of the waste oil piles would be less than those with nutrients. However the control pile showed as high bioactivity as the others. It was not N limited. It looks as if the degrading bark has released nutrients and there was only about a month delay in the onset of oil degradation in the control. During the first month also the bark has very likely needed extra nutrients to be degraded. After mineralization the bark released lot of nutrients (see p. 13, Table 2) available for oil degrading microbes. No nutrients leached with water from the system. Some loss of nitrogen may be due to denitrification (see below).

NH_4-N and NO_3-N levels were high in pile 5 receiving two doses of fertilizer. The rate of oil hydrocarbon degradation was not affected by this higher dose. NH_4-N dropped between day 80 and 95, which matches increase in NO_3-N approximately. This indicates that NH_4-N was nitrified. To some extend it was immobilized in biomass, which fits with reduced biodegradation at this time.

The delay itself may be very crucial in our climate. Therefore nutrient addition

seems to be essential for a rapid process. According to the results of this project the amendment would be somewhat less than 1 kg (nitrogen rich fertilizer) per m^3 compost matrix (approx. 0,15-0,2 g N kg^{-1} compost matrix). The actual doses have to be estimated case by case. One can also remember that overdosing in reasonable amounts does not harm the process. On the other hand, excessive fertilizer applications have been demonstrated to inhibit bioremediation of petroleum hydrocarbons. It is recommended (Shell Research Center 1994, personal communication) that e.g. ammonium nitrate should not exceed 5 g l^{-1} soil solution.

Apparently the soluble nutrients were used up quite fast during the first couple of months. A constant nitrogen mineralisation from the bark is probably going on during the period. Part of the nitrogen added seems to have been denitrified. This may be due to anaerobic microsites in compost matrix, which are an important part of an essentially aerobic system making anaerobic denitrification possible.

According to the measured denitrification rates under completely aerobic conditions (average 5 ng N g^{-1} h^{-1}) a loss of N of 0.12 mg N kg^{-1} (dry soil) d^{-1} could have occured. Under completely anaerobic conditions (average 1000 ng N g^{-1} h^{-1}) a loss of 24 mg N kg^{-1}(soil) d^{-1} could occur. The true oxygen conditions in the compost piles were not measured but CO_2 concentrations up to 35 % in the soil gas air indicated that the oxygen concentrations would have been fairly low in between the turning of the piles (see p. 20). The true loss of nitrogen due to denitrification was thus between 0.12 and 24 mg N kg^{-1} (dry soil) d^{-1}. The nitrogen addition was 117 mg NO_3^--N kg^{-1} and 138 mg NH_4^+-N kg^{-1}.

6.4 Moisture

According to the preliminary plan moisture was one of the parameters to be adjusted in the piles. Due to the small volume of lube oil contaminated soil and the numerous other parameters to be tested the effect of moisture was not systematically measured. Water was retained by the use of protecting tarpaulins. The main reason to do so was that the soil contained on average 20 % of water. Also the bark was very wet (dry weight 41 %). There was therefore not any practical reason to test extra watering in the five lube oil piles which were already too small to be divided for moisture experiments.

However watering was tested in diesel oil piles in the later phase (in August) of the study. The water content of the test pile was increased about 4 %. Extra water had no significant effect on biodegradation. Increase in moisture content was obviously so small that it could not have major effect on microbial avtivity.

The water content may affect the oxygen availability. Oxygen diffuses slowly through water and a more moist soil may become anaerobic faster than a less moist one.

The minimum and optimum moisture content for composting cannot be estimated from this work. It looks anyhow obvious that in many cases soils in Finland contain enough water for proper bioactivity. However depending on weather conditions and soil types situation can vary a lot case by case. In this work any greater losses of moisture could not be accepted. Therefore it is wise to cover the piles to prevent evaporation. Covering also prevents the piles packing during heavy rain fall.

6.5 Inocula

Two commercial inocula were tested. The result in this as in many other experiments was that the use of external microbes had no significant effect on the process. The natural population in soil is usually able to degrade oil compounds. It is more important to create suitable conditions for these indigenous bacteria. Inocula may be beneficial in certain special cases (single recalcitrant compouds) but for petroleum hydrocarbons they are only an extra cost and give no benefit. It is also important to know that none of the introduced organisms were reisolated by traditional plating techniques.

6.6 Reaching the clean-up target

The aim of the study was to find out how efficient method composting is and how low a mineral oil concentration could be reached during one growth season. In the gasoline contaminated soil piles 100 mg kg^{-1} dw was reached in about two months. However the result does not tell the real potential of composting, because of the low initial oil hydrocarbon concentrations. For the lube oil contaminated soil final concentrations (after five months processing) were about 700 mg kg^{-1} dw. Almost 70 % reduction was reached. Albeit, more heavily contaminated soil is needed to make conclusions about the efficiency of the short time composting to reach the strict concentration limits suggested for the dumping of the contaminated soils.

In spite of the low initial oil hydrocarbon concentrations the result can be extended to more polluted soil. The findings of British researchers (ref. Shell Research Center 1994) are that the same degradation pattern for the oil hydrocarbons can be obtained even with concentrations 10 000 - 50 000 mg kg^{-1}

dw. Very high concentrations naturally mean prolonged duration of composting to reach the target limits.

The aim was to reuse the purified soil at a landfill. This was done already in August 1993 due to the space problem (to get more space for the municipal sludge composting on the field). Part of the "cleaned" gasoline contaminated soil was moved away and used as cover material on the garbage at the dumping site nearby.

The piles were turned and analyzed in spring 1994 after they had thawed out. The follow-up will be continued by WERI for as long as it is practically possible.

6.7 Costs of composting

The actual cost of this experimental full scale composting is around 70-80 FIM per m^3 contaminated soil. The sum excludes excavation and transportation of the soils. It also excludes the price of the bulking agent (spruce bark) which in this case was obtained for transportation costs. The organizing work (planning, supervision, etc.) is not covered either. On the other hand the expenses include a lot of extra chemical, physical and microbiological analyses (worth ca. 30 000 FIM) which are not all needed for routine composting. The real cost i.e. the current price considering the same kind of circumstances (ca. 1000 m^3 contaminated soil) without earthwork would probably be of the order of 100-120 FIM per m^3. In this case excavation and transportation cost slightly more than the actual composting work. The total cost was approx. 250 FIM per m^3. This is the sum estimated in 1993 also by other composting contractors for a similar type of case.

Composting can be regarded as fairly moderate price method for remediating oil contaminated soil.

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YHTEENVETO

Öljyalan keskusliitto (ÖKL) tilaajana ja vesien- ja ympäristöntutkimuslaitos (VYL) suorittajana ovat toteuttaneet projektin, jossa jälkimmäinen on selvittänyt biologisten menetelmien soveltuvuutta saastuneen, käytöstä poistettavan huoltoasemakiinteistön maa-aineksen käsittelemiseksi. Esiselvityksen pohjalta käytännön saneeraustyö tehtiin kompostoiden. Tutkimuksen tarkoituksena oli selvittää, kuinka nopeasti jäteöljyllä likaantunutta maata voidaan puhdistaa yhden kesäkauden aikana. Projektiin liittyy jatkossa tehtävä kunnostusohjeiden laadinta.

Kolmella Helsinki-alueen huoltoasemakiinteistöllä, joilla oli tiedossa aikojen kuluessa tapahtunutta maa-aineksen likaantumista poltto- ja voiteluaineilla (ei hallitsemattomia vuotoja) selvitettiin maaperän likaantumisaste ja maan määrä. Selvitys tehtiin maaperä-, pohjavesi- ja huokoskaasuanalyysein. Likaaviksi aineksiksi osoittautuivat jäteöljyt, bensiini ja dieselöljy.

Likaantunut maa (yht. n. 800 m^3) kaivettiin ylös ja kuljetettiin Nurmijärven kunnan Metsä-Tuomelan jäteaseman kompostointikentälle. Maat seulottiin ja kompostoitiin kesän 1993 aikana aumoissa, joissa apuaineina käytettiin kuorikettä ja ravinteita. Koemielessä lisättiin myös mikrobeja. Aumojen pH säädettiin kalkilla ja ne sekoitettiin ruuvityyppisellä sekoittimella. Aumat peitettiin ja niistä otettiin näytteitä sekoitusten yhteydessä kemiallisia ja mikrobiologisia analyysejä varten.

Maa-ainesten öljytuotteiden määrät alenivat viiden kompostointikuukauden aikana selvästi. Alkuperäinen jäteöljypitoisuus aleni kahdella kolmanneksella, 2400 mg:sta 700 mg:an kilossa kuivaa maata. Hajoamisnopeus oli suunnilleen sama riippumatta alkupitoisuuksista. Nopeinta hajoaminen oli kahden ensimmäisen kuukauden aikana. Syksyllä säiden viiletessä ja helposti hajoavien aineiden vähetessä bioaktiivisuus aleni selvästi.

Ravinteiden lisääminen ei vaikuttanut juurikaan lopputulokseen, mutta se kiihdytti hajomista prosessin alussa. Suomen ilmastoa ajatellen tästä on etua. Mikrobilisäyksillä ei ollut merkitystä tulokseen. Aumojen peittäminen osoittautui tarpeelliseksi haih-dunnan ja sateitten aikana tapahtuvan aumojen tiivistymisen estämiseksi.

Varsinaisen kompostointityö maksoi noin 70-80 FIM/m^3 likaista maata. Todelliset markkinahintaiset kustannukset saman tapaiselle kompostoinnille (so. noin 1000 m^3 maata) sisältäen maan kaivuun ja kuljetuksen olisivat noin 250 FIM/m^3. Hinta on samaa suuruusluokkaa kuin on arvioitu toisaalla koskien vastaavanlaista urakkaa. Kompostointia voidaankin kustannuksiltaan pitää varsin edullisena maansaneeraus-tapana Suomessa. Nyt saatu hyvä tulos kannattaa jatkossa varmentaa voimakkaasti saastuneilla massoilla. Kompostoinnin edut voidaan silloin todentaa myös ehdotet-tujen uusien ja tiukempien saastuneen maan käsittelynormien valossa.

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Biological treatment of oil contaminated soil has received much attention also in Finland during last years. However, research is needed to ensure that new technologies are implemented in a safe and reliable way under cold climatic conditions. During 1993 the biotreatability of oil-contaminated soil from gasoline stations was investigated. The goal of the field-scale study was to test how fast lubrication oil can be composted during one Finnish summer season.

The contaminated soil was excavated from three different service stations in the Helsinki metropolitan area and transported to an asphalted compositing area. The soils were sieved and composted in piles with bark and nutrients. Two different commercial microbial inocula were added into the piles. The piles were aerated by turning with a tractor-drawn screw-type mixer and sampled for chemical and microbiological analyses after each mixing.

The result shows that there was a distinct decline in the mineral oil concentrations during the composting. Two thirds of the original oily material was degraded during 5 months. The pattern was about the same in piles with low and high starting concentrations. External microbes had no major effect on the process. According to the total cost (ca. 250 FIM/m^3) composting can be regarded as fairly low price method for remediating oil contaminated soil.

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